# ANALYSIS OF FROZEN CHICKEN MEAT USING DIFFERENTIAL SCANNING CALORIMETRY

Danica M. Savanović[*1], Radoslav D. Grujić[2], Jovo M. Savanović[3], Snježana U. Mandić[1], Slađana M. Rakita[4]

[1] University of Banja Luka, Faculty of Technology, 78 000 Banja Luka, Vojvode Stepe Stepanović 73, Republic of Srpska, Bosnia and Herzegovina
[2] University of East Sarajevo, Faculty of Technology, 75 400 Zvornik, Karakaj 34A, Republic of Srpska, Bosnia and Herzegovina
[3] Meat industry DIM-DIM, Laktaši, 78 000 Banja Luka, Republic of Srpska, Bosnia and Herzegovina
[4] University of Novi Sad, Institute of Food Technology, 21000 Novi Sad, Bulevar cara Lazara 1, Serbia

*Corresponding author:
Phone: +387 65 995 374
Fax: +387 51 434 351
E-mail address: danica.savanovic@tf.unibl.org

**ABSTRACT:** The paper analyses the effect of cooling/heating rate of chicken meat (*Pectoralis major*) on the crystallization temperature ($T_c$on, $T_c$, $T_c$end), melting temperature ($T_m$on, $T_m$, $T_m$end), crystallization enthalpy ($\Delta H_c$) and melting enthalpy ($\Delta H_m$). Chicken meat samples were scanned by differential scanning calorimetry (DSC) at five rates (2, 5, 10, 15, 20 °C/min), from 20 °C to -40 °C, and then from -40 °C to 20 °C. The results of the statistical analysis show that the fastest cooling rate (20 °C/min) significantly ($p < 0.05$) affects the mean enthalpy value (-202.87 J/g) compared to other analysed rates. The cooling/heating rate affects the crystallization temperature ($T_c$on, $T_c$, $T_c$end) and melting temperature ($T_m$on, $T_m$, $T_m$end) ($p < 0.05$). The heating rate of chicken meat highly correlates with $T_m$, $T_m$end and $\Delta T_m$ (the correlation coefficients were 0.993, 0.998 and 0.998, respectively).

**Key words:** *DSC, chicken meat, crystallization, melting, freezable water, unfreezable water*

## INTRODUCTION

Differential scanning calorimetry (DSC) is a technique commonly used for the analysis of phase transition and thermo-physical properties of food (Zaidul et al., 2008; Dahimi et al., 2014; Tolstorebrov et al., 2014; Karthikeyan et al., 2015). The method involves scanning the analyzed sample and the reference sample at a preset cooling/heating rate. On the curve obtained during the DSC analysis, each difference in heat energy is recorded as a certain peak (Schubring, 1999; Zielbauer et al., 2016). The DSC generated thermogram indicates the change of the thermal flow depending on the temperature. In this way, it is possible to obtain data on the amount of energy which is released or received during phase transition (Tomaszewska-Gras, 2013; Tolstorebrov et al., 2014).

DSC can be used for the determination of the thermo-physical properties and their changes depending on temperature and food structure. Previous researches have been devoted to the determination of properties of fishery products (Schubring, 1999), fresh meat (Voutila et al., 2009; Castro-Giráldez et al., 2014; Savanović et

al., 2016; Savanović et al., 2017), bread (Ribotta and Le Bail, 2007a), bread dough (Matuda et al., 2011), partially baked bread (Ribotta and Le Bail, 2007b) and fruit (Falcao-Rodrigues et al., 2007; Syamaladevi et al., 2010). DSC is commonly used to determine the phase transitions such as crystallization and melting in food (Hamdami et al., 2004; Yilmaz and Karakaya, 2009; Matuda et al., 2011; Tolstorebrov et al., 2014).

Crystallization of water during freezing, as well as melting of ice which occurs in the process of thawing, are the most frequent phase transitions in food (Grujić et al., 1993; Kiani and Sun, 2011; Tolstorebrov et al., 2014). Freezing is one of the oldest and most commonly used food preservation methods. Compared to other preservation methods, in frozen foods, the main ingredients and labile food components are best preserved. Changes of the proteins of meat and fish during freezing do not cause great quality losses during long-term storage of these frozen products (Soyer et al., 2010). Freezing and frozen storage are widely performed methods in meat industry in order to preserve nutritional and sensory properties of meat products (Soyer et al., 2010; Bueno et al., 2013; Xanthakis et al., 2013). Physico-chemical and sensory characteristics of meat may be altered after freezing and thawing, and, due to this, these processes have a large effect on the quality of meat (Petrović et al., 1993; Grujić et al., 2003; Bueno et al., 2013).

Therefore, studying the effects of freezing and frozen storage on physical and chemical changes in meat is very important for meat processing (Bertram et al., 2007). DSC is a method suitable for measuring the freezing and melting properties of food, for example the freezing, melting, and the glass transition temperatures. DSC analysis can be performed with different scanning rates. However, there are only few studies in the literature which compare DSC thermograms with respect to different cooling and heating rate of food. The aim of this paper was to define the temperatures of crystallization, melting and glass transition of chicken meat (*Pectoralis major*), using differential scanning calorimetry (DSC), at different cooling/heating rates.

## MATERIALS AND METHODS

### Standard procedure

The study investigated fresh post-rigor chicken meat (*Pectoralis major*), purchased at a slaughter house.

Differential scanning calorimetry thermograms were obtained using a differential scanning calorimeter DSC (204 F1 Phoenix, Netszch). The samples (14±2 mg) were weighed into 25 μl capacity aluminum pans. After that, the pans were hermetically sealed. An equal empty pan was used as a reference sample during the experiments. Cell calibration was carried out according to the DSC recommendations provided by the manufacturer. The flow rate of purge nitrogen gas was 20 ml min$^{-1}$.

The temperatures of onset crystallization ($T_c$on), peak crystallization ($T_c$), end crystallization ($T_c$end) and enthalpy of crystallization ($\Delta H_c$) were measured from the cooling curves and analyzed for the crystallization process. The following temperatures were analyzed for the melting process: the temperatures of onset melting ($T_m$on), peak melting ($T_m$) and end melting ($T_m$end). The melting temperature interval was computed as the width of the melting peak ($\Delta T_m = T_m$end-$T_m$on) and the crystallization temperature interval was calculated as the width of the crystallization peak ($\Delta T_c = T_c$on-$T_c$end). The enthalpy of melting $\Delta H_m$ (J/g) was calculated as the area between the melting curve and the baseline. The DSC thermogram analysis and the determination of thermo-physical properties of chicken meat were conducted using the Proteus software, version 6.1.0 (NETZSCH–Gerätebau GmbH, Germany). The glass transition temperature ($T_g$) was calculated from the DSC melting curves, using Proteus software.

### The influence of the scanning rate on thermo-physical properties of chicken meat

The samples of chicken meat were cooled and heated at five rates (2, 5, 10, 15, 20 °C/min). First of all, the samples were

equilibrated (20 °C for 5 min) and cooled to -40 °C at a selected rate. After an iso-thermal phase (-40 °C for 5 min), the samples were heated at the identical rate to 20 °C. The analysis of all the samples was repeated three times.

### Determination of freezable water

The percentage of freezable water (*FW*) in chicken meat was determined according to Eq.1 (Xanthakis et al., 2013):

$$FW\ (\%) = \frac{\Delta H_m \cdot 100}{H_f \cdot m_s} \qquad (1)$$

Where *FW* is the percentage of freezable water, $\Delta H_m$ is the enthalpy of melting (J/g), $H_f$ represents theoretical enthalpy of melting ice at 0 °C (333.50 J/g) and $m_s$ is the mass of the sample. The percentage of unfreezable water (*UFW*) was deter-mined as the difference between the total water percentage and percentage of fre-ezable water (Simmons et al., 2012). The total water content of chicken meat was determined by drying at 105±2 °C to con-stant mass (AOAC, 2006).

### Statistical analysis

The obtained results are expressed as the mean values and standard deviations of three measurements. One factor analysis of variance (ANOVA) was done by ap-plying the IBM SPSS Statistics for Win-dows, version 22.0 (Armonk, NY, United States). The Duncan's Post-hoc test, at a significance level of 0.05, was used for the comparison of treatment means and crea-ting statistically homogeneous groups.

## RESULTS AND DISCUSSION

### The influence of the scanning rate on the crystallization processes in chicken meat

Meat is a complex system including va-rious compositions and structures. There-fore, the characterization of meat can be challenging. Many analytical methods have been used to study food, including DSC. This technique can be used to investigate thermal transition of various food products (Schubring, 1999; Toma-szewska-Gras, 2013; Dahimi et al., 2014).

Figure 1 presents crystallization curves of chicken meat obtained using different cooling rates (2, 5, 10, 15 and 20 °C/min). For the tested chicken meat samples, various cooling rates caused diverse peak shapes, different sizes and different tem-peratures. As seen in Figure 1, the width of crystallization peaks decreased with the decrease in cooling rate, which led to a significant (p<0.05) change of the crystal-lization enthalpy ($\Delta H_c$) of chicken meat. The mean values of the crystallization enthalpy ($\Delta H_c$) for the rate 20 °C/min (-202.87 J/g) was markedly (p<0.05) different from the values of crystallization enthalpy for other rates (Table 1). Si-milarly, Tomaszewska-Gras (2013) ob-served a decrease in the width of crys-tallization peaks of milk fat with a decrease in cooling rate.

Different transition temperatures of crys-tallization for chicken meat were recorded for various cooling rates (2, 5, 10, 15 and 20 °C/min). The study did not find any significant (p>0.05) differences between the values of the onset crystallization temperature ($T_c$on), as well as the tempe-rature of peak crystallization ($T_c$) for the cooling rates 2, 5 and 20 °C/min (Table 1). The temperature at which the process of crystallization ($T_c$on) in chicken meat begins, for the rates 10 and 15 °C/min were -18.90 °C and -17.77 °C, respecti-vely, which is significantly different (p<0.05) from the other rates. Similarly, the mean values of peak temperatures ($T_c$) for chicken meat, for the rate 10 and 15 °C/min, were -19.27 °C and -18.30 °C, respectively, being significantly different (p<0.05) from the peak temperatures detected in DSC curves at 2, 5 and 20 °C/min cooling rates. The mean values of end crystallization temperature ($T_c$end) for chicken meat significantly (p<0.05) changed with the increase in the cooling rate (Table 1). The mean value of $T_c$end of chicken meat was -14.97 °C (rate 2 °C/min), -22.20 °C (rate 15 °C/min) and -18.73 °C (rate 20 °C/min). It was observed that the cooling rate had a significant (p<0.05) effect on the crystallization temperature interval ($\Delta T_c$), which in-creased with the increase in the cooling rate from 1.37 °C (rate 2 °C/min) to 5.97 °C (rate 20 °C/min) (Table 1).
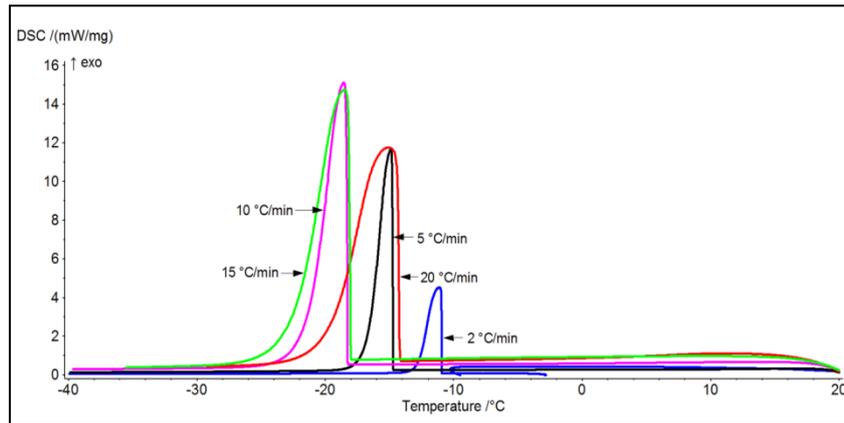
Figure 1. DSC crystallization curves of chicken meat at different cooling rates
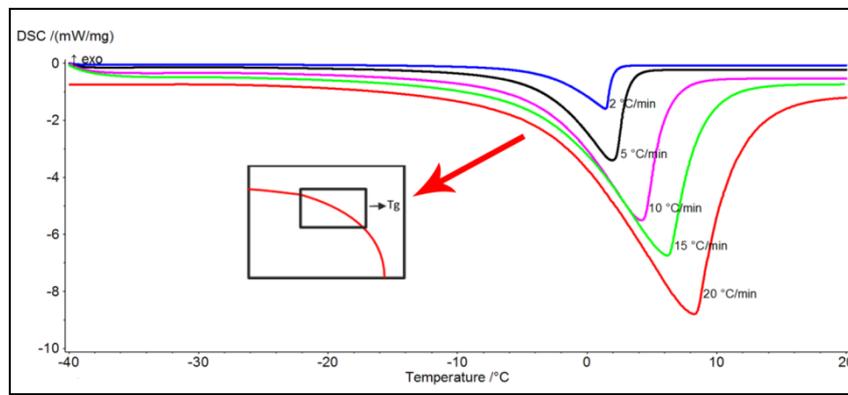(2, 5, 10, 15 and 20 °C/min)



Figure 2. DSC melting curves of chicken meat at different heating rates (2, 5, 10, 15 and 20 °C/min)
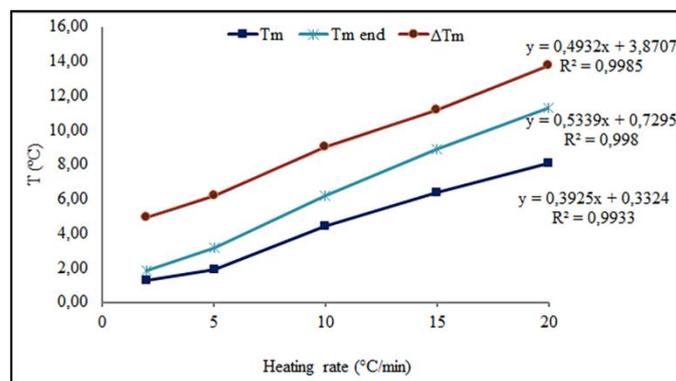


Figure 3. Relationship between heating rate (2, 5, 10, 15 and 20 °C/min) and $T_m$; $T_m$end; $\Delta T_m$
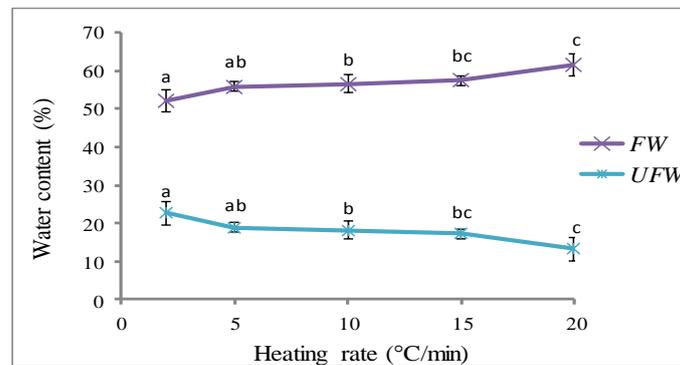


Figure 4.The content of freezable (*FW*) and unfreezable water (*UFW*) in chicken meat

**Table 1.**
Crystallization temperatures and enthalpy for chicken meat in relation to cooling rate

| Rate (°C/min) | Crystallization (from +20 °C to -40 °C) | | | | |
|---|---|---|---|---|---|
| | $\Delta H_c$ (J/g) | $T_c$on (°C) | $T_c$ (°C) | $T_c$end (°C) | $\Delta T_c$ (°C) |
| 2 | -178.13[b] ±6.95 | -13.60[b] ±1.70 | -13.70[b] ±1.60 | -14.97[c] ±1.25 | 1.37[a] ±0.45 |
| 5 | -185.67[b] ±2.90 | -15.10[b] ±0.40 | -15.40[b] ±0.50 | -17.80[b] ±1.10 | 2.70[b] ±0.70 |
| 10 | -183.33[b] ±6.53 | -18.90[a] ±1.21 | -19.27[a] ±1.15 | -22.00[a] ±1.30 | 3.10[b] ±0.10 |
| 15 | -187.67[b] ±6.08 | -17.77[a] ±1.27 | -18.30[a] ±1.21 | -22.20[a] ±1.01 | 4.43[c] ±0.25 |
| 20 | -202.87[a] ±0.61 | -12.77[b] ±1.24 | -14.23[b] ±0.87 | -18.73[b] ±0.85 | 5.97[d] ±0.42 |

*Data are expressed as mean ± standard deviation.*
[a,b]*Mean values in the same colon followed by different online letters indicate significant difference (p<0.05);*
*$\Delta H_c$ - enthalpy of crystallization; $T_c$on - temperature of onset crystallization; $T_c$ - temperature of peak crystallization; $T_c$end - temperature of end crystallization; $\Delta T_c$ - width of crystallization peak*

**Table 2.**
Melting temperature and enthalpy for chicken meat, in relation to heating rate

| Rate (°C/min) | Melting (from -40 °C to +20 °C) | | | | | |
|---|---|---|---|---|---|---|
| | $\Delta H_m$ (J/g) | $T_g$ (°C) | $T_m$on (°C) | $T_m$ (°C) | $T_m$end (°C) | $\Delta T_m$ (°C) |
| 2 | 173.67[a] ±10.03 | -6.90[a] ± 0.26 | -3.03[a] ±0.15 | 1.30[a] ±0.10 | 1.87[a] ±0.06 | 4.90[a] ±0.10 |
| 5 | 186.17[ab] ±4.16 | -6.44[ab] ± 0.62 | -3.03[a] ±0.15 | 1.93[b] ±0.06 | 3.17[b] ±0.06 | 6.20[b] ±0.17 |
| 10 | 188.53[b] ±7.96 | -6.03[bc] ± 0.15 | -2.80[ab] ±0.00 | 4.40[c] ±0.44 | 6.20[c] ±0.36 | 9.00[c] ±0.36 |
| 15 | 191.83[bc] ±4.27 | -5.93[bc] ± 0.23 | -2.23[c] ±0.21 | 6.37[d] ±0.15 | 8.90[d] ±0.20 | 11.13[d] ±0.25 |
| 20 | 205.20[c] ±10.12 | -5.40[c] ± 0.18 | -2.50[bc] ±0.44 | 8.07[e] ±0.25 | 11.27[e] ±0.35 | 13.77[e] ±0.15 |

*Data are expressed as mean ± standard deviation.*
[a,b]*Mean values in the same colon followed by different online letters indicate significant difference (p<0.05);*
*$\Delta H_m$ - enthalpy of melting; $T_m$on - temperature of onset melting; $T_m$ - temperature of peak melting; $T_m$end - temperature of end melting; $\Delta T_m$- width of melting peak; $T_g$ -glass transition temperature*

The first ice crystals start to form at a temperature which is defined as the initial freezing point. Marini et al. (2014) observed considerably different initial freezing temperatures and the end points of freezing for mechanically deboned chicken meat, chicken sausages (frankfurter type) and mortadella sausage (bologna type). In their study, the following values of the initial freezing temperature and the end freezing temperature were measured: -0.43 °C and -4.46 °C (mechanically deboned chicken meat), -2.49 °C and -9.71 °C (frankfurter sausages), -4.46 °C and -10.14 °C (bologna type sausage). Meat is a complex mixture and many different reactions occur in it during processing and storage (Zhu et al., 2006), which is not the case with clear substances. The initial values for a clear substance should always be the same, despite various scanning rates. For the analyzed food samples, different cooling rates caused disparate flows of crystallization, and the obtained peaks have unequal sizes and shapes, as well as different temperatures (Tomaszewska-Gras, 2013). The factors which affect the freezing process in food are the following: the content of water, properties and contents of other food ingredients, soluble and insoluble solid substances, specific heat, enthalpy and the transfer of

mass and energy (Jie et al., 2003). The insoluble components such as fat and insoluble proteins will not affect the change of freezing point, but the soluble components, such as heterogeneous sugars, ions and acids and soluble proteins, can cause the depression of freezing point. Components that have high molecular masses, such as protein and starch, contribute insensibly to the mole fraction of solutes while the molecules with small molecular masses such as sugars, ions and acids, contribute noticeably (Miles et al., 1997).

## The influence of scanning rate on the melting processes in chicken meat

Figure 2 presents the melting curves of chicken meat obtained at different heating rates (2, 5, 10, 15 and 20 °C/min), following the cooling of samples with the same rates to the final temperature of -40 °C. As shown in Figure 2, heating of chicken meat at different rates (from 2 to 20 °C/min) caused various changes in the process of ice melting. The shape of the curves, as well as the width of melting peaks changed depending on the heating rate. The crystallization curves have narrower peaks than the curves of melting, as it can be seen in Figure 1 and 2. This indicates that the process of thawing in meat occurs more slowly than freezing. The temperature interval of thawing is wider than that of freezing and the changes that take place during thawing are more gradual in comparison to freezing, as has already been observed (Karthikeyan et al., 2015). Similarly to the crystallization process, the size and the position of peaks and the shape of the melting curves changed with various heating rates.

The values of the enthalpy of melting ($\Delta H_m$) significantly (p<0.05) increased with the increase in the heating rates from 2 °C/min to 20 °C/min. For the heating rate 2 °C/min, the value of the enthalpy of melting was 173.67 J/g, and 205.20 J/g for the heating rate 20 °C/min (Table 2).

An important feature in the study of food processing and different aspects of food stability is glass transition. The glass transition approach can be used as a parameter for the estimation of water mobility and food stability. Below the glass transition temperature ($T_g$) food is quite stable (Ostojić et al., 2014). Glass transition is a second-order phase transition, a property of food that occurs in a distinctive glass transition temperature interval (Figure 2). The mean values of glass transition temperature ($T_g$) for chicken meat significantly (p<0.05) changed with the increase in the heating rate (Table 2), ranging from -6.90 °C for the rate 2 °C/min to -5.40 °C for the rate 20 °C/min. Akkose and Aktas (2009) found that the $T_g$ value for rainbow trout was -13 °C and authors considered that -13 ºC could be an indicator of usability instead of -18 °C. Different chemical reactions can cause deterioration of frozen food. The $Tg$ concept is a credible measure for evaluation food stability at sub-freezing temperatures (Akkose and Aktas, 2008).

Different transition temperatures of melting for chicken meat were recorded with various heating rates (2, 5, 10, 15 and 20 °C/min). In this research, the values of the onset melting temperature ($T_m$on) gradually increased from -3.03 °C to -2.50 °C with increase in heating rate from 2 to 20 °C/min. Further, significant (p<0.05) differences between mean values of peak melting ($T_m$) and end melting temperature ($T_m$end) of chicken meat were observed for all the heating rates. For the rate 2 °C/min, the peak melting ($T_m$) temperature was 1.30 °C, and for the rate 20 °C/min 8.07 °C. Moreover, the end melting temperature ($T_m$end) significantly increased (p<0.05) from 1.87 °C to 11.27 °C, with increase in heating rate from 2 to 20 °C/min. Analogously to the process of crystallization, reduction of the heating rate caused a significant (p<0.05) decrease in the width of the melting peak ($\Delta T_m$) for chicken meat, and the width of the melting peak ($\Delta T_m$), in which the melting process of water takes place, increased with the increase of the heating rate (Table 2).

As it can be seen in Figure 3, there are linear relationships between the heating rate and the temperature of peak melting ($T_m$), the temperature of end melting ($T_m$end) and the melting temperature interval ($\Delta T_m$) for chicken meat. The re-

sults of the statistical analysis show a high correlation between the heating rate and $T_m$ ($R^2$=0.993), $T_m$end ($R^2$=0.998) and $\Delta T_m$ ($R^2$=0.998) for chicken meat.

Understanding of thermo-dynamic properties (e.g. initial freezing temperature, frozen water content, specific heat and enthalpy) at low temperatures is very important from the aspect of process design, the choice of appropriate equipment, the assessment of freezing and thawing time, and operating expenses. The most important factors affecting the thermal and physical characteristics of food products are their composition and temperature (Matuda et al., 2011).

**The influence of the scanning rate on the content of freezable and unfreezable water in chicken meat**

Different types of meat have different contents of water that can undergo the phase transition. In regard to its behavior during freezing, the water present in meat can be divided into two types: freezable and unfreezable water. Only the first type of water crystallizes into ice (Ding et al., 2015). It is arbitrarily chosen that 100% of the freezable water will freeze at a temperature of -40 °C. Accordingly, the quantity of unfrozen water at the same temperature (-40 ºC) is known as "unfreezable water" (Hamdami, 2004; Fasina, 2012; Tolstorebrov, 2014). The unfreezable water is bound water, while the freezable water represents the free water fraction of the total water content in a product.

Analogously to the enthalpy of melting, the mean values of freezable water content and unfreezable water content significantly differ (p<0.05) for the heating rates from 2 to 20 °C/min. A significant (p<0.05) increase in the freezable water content was recorded in chicken meat, from 52.07% for the rate 2 °C/min to 61.53% for the rate 20 °C/min, and a significant (p<0.05) decrease in the unfreezable water content from 22.69% for the rate 2 °C/min to 13.23% for the rate 20 °C/min (Figure 4). Tolstorebrov et al. (2014) reported that the amount of unfreezable water calculated by the DSC melting endotherm integration for the scanning rate of 5 °C/min for Atlantic Salmon, Cod, Herring, Mackerel and Rain-

bow Trout was in the range between 5.1% to 8.6%.

Thermo-physical properties of meat products depend on their chemical composition, especially the water content. Deteriorative reactions in meat are the main cause for the decrease in quality during storage, and the primary reason for product spoilage. These reactions in the meat tissues are normally inhibited by decreasing the product temperature, which results in the increase of shelf life. The length of a high quality shelf life (the general trend) increases exponentially with the temperature decrease from -5.0 °C to -30.0 °C (Tolstorebrov et al., 2014). Acceptable storage is achieved at -18.0 °C and below.

The most significant change that occurs during freezing of food is the crystallization of water into ice (Xanthakis et al., 2013). The modeling of the freezing process contributes to understanding the complex changes that are caused by the temperature changes and modification in the content and attainability of water in a food product. In addition, it can help to identify stability of food during preservation, as well as in selection of an appropriate processing condition (Rahman, 2006).

## CONCLUSIONS

Differential scanning calorimetry (DSC) can be used to measure the enthalpies and temperatures of phase transitions in chicken meat. The study showed that the cooling rate from 2 to 20 °C/min affected the enthalpy ($\Delta H_c$) and the end temperatures ($T_c$end) of the crystallization process (p<0.05). The effect of the heating rate on the temperatures of the melting process ($T_m$on, $T_m$, $T_m$end) for chicken meat was significant (p<0.05). The values of the enthalpy of melting, as well as the freezable water content in chicken meat were significantly (p<0.05) different for the heating rates from 2 to 20 °C/min. The width of the crystallization temperature interval ($\Delta T_c$), in which water crystallization takes place, increased with the increasing in the cooling rate (p<0.05), from 1.37 °C (rate 2 °C/min) to 5.97 °C (rate 20 °C/min). Similarly, the width of the melting peak ($\Delta T_m$) increased (p<0.05) with increasing

heating rate, from 4.90 °C (rate 2 °C/min) to 13.77 °C (rate 20 °C/min).

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## АНАЛИЗА СМРЗНУТОГ ПИЛЕЋЕГ МЕСА ДИФЕРЕНЦИЈАЛНОМ СКЕНИРАЈУЋОМ КАЛОРИМЕТРИЈОМ

Даница М. Савановић*[1], Радослав Д. Грујић[2], Јово М. Савановић[3], Сњежана У. Мандић[1], Слађана М. Ракита[4]

[1] Универзитет у Бања Луци, Технолошки факултет, 78 000 Бања Лука, Војводе Степе Степановић 73, Република Српска, Босна и Херцеговина
[2] Универзитет у Источном Сарајеву, Технолошки факултет, 75 400 Зворник, Каракај 34А, Република Српска, Босна и Херцеговина
[3] Месна индустрија ДИМ-ДИМ, Лакташи, 78 000 Бања Лука, Република Српска, Босна и Херцеговина
[4] Универзитет у Новом Саду, Научни институт за прехрамбене технологије у Новом Саду, 21000 Нови Сад, Булевар цара Лазара бр. 1, Србија

**Сажетак**: Рад анализира утицај брзине хлађења/загревања пилећег меса (*Pectoralis major*) на температуре кристализације ($T_c$on, $T_c$, $T_c$end), температуре топљења ($T_m$on, $T_m$, $T_m$end), енталпију кристализације ($\Delta H_c$) и енталпију топљења ($\Delta H_m$). Узорци пилећег меса скенирани су диференцијалном скенирајућом калориметријом (DSC), на пет брзина (2, 5, 10, 15, 20 °C/min од 20 °C до -40 °C, затим од -40 °C до 20 °C. Резултати статистичке анализе показују да највећа брзина хлађења (20 °C/min) значајно (p<0,05) утиче на средњу вредност енталпије (-202,87 J/g) у односу на друге анализиране брзине. Брзина хлађења/загревања утиче на температуре кристализације ($T_c$on, $T_c$, $T_c$end) и температуре топљења ($T_m$on, $T_m$, $T_m$end) (p<0,05). Брзина загревања пилећег меса је у корелацији са $T_m$, $T_m$end и $\Delta T_m$ (коефицијенти корелације су били 0,993, 0,998 и 0,998, респективно).

**Кључне речи**: *DSC, пилеће месо, кристализација, топљење, смрзнута вода, несмрзнута вода*